**CENTRAL REGIONAL LABORATORY**

Data Checklist

Data Set AER 2101 0052 Cheshire Monitoring

☑ Chain-of-Custody

☑ Analysis Request Form(s)*

☐ Sample Tags

☑ Transmittal Report w/signatures of the following

- Analyst(s)
- Peer reviewer
- Data Management Coordinator

* Analysis Request Forms provide the data user a means to connect sample numbers with sampling locations.

Prepared by: Sylvia Griffin 8/10/2001
Data Management Coordinator

Rev. 6/12/00

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CENTRAL REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

Date: AUG 10 2001

Subject: Review of Region 5 Data for **Cheshire Monitoring Study**

From: **John V. Morris, Chemist**
Region 5 Central Regional Laboratory

To:

Attached are the results for: **Cheshire Monitoring Study**

CRL data set number: **20010052**

Samples analyzed for: **Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Magmesium, Manganese, Nickel and Selenium**

Results are reported for sample designations: **2001AH02S01, 2001AH02D01, 2001AH02S02 and 2001AH02S03**

AUG 10 2001 / /

Data Management Coordinator and Date Received

Date Transmitted: AUG 10 2001 / /

Please have the U.S. EPA Project Manager/Officer complete the Customer Satisfaction Survey, attached, or call the CRL Sample Coordinator at 3-1226.

Please sign and date this form below and return it with any comments to:

Sylvia Griffin
Data Management Coordinator
Region 5 Central Regional Laboratory
ML-10C

/ /

Received by and Date

Comments:

# Central Regional Laboratory, RMD, Region 5
# Customer Satisfaction Survey

The purpose of this survey is to collect information from you about your recent experience with analytical services received from the Region 5 Central Regional Laboratory (CRL). This survey is divided into 4 sections. Please fill out the information in each section as requested. Then in Section C, supply your name and contact information, and submit the form as directed at the end of the survey.

## Section A -- Sample Requests

Please respond to the following questions as accurately as possible. If you have additional comments beyond the space provided, please send them to George Schupp, CRL Sample Coordinator, at ML-10C (See Form Submission).

1. What is your CRL Data Set Number(s) [i.e., the 8-digit number beginning with the 4-digit FY and followed by a 4 digit number]? (Eg.:20010099) _____

2. How easy was it to schedule samples? :

   Easy: _____ Difficult: _____

3. If not "Easy", please provide a brief explanation:

## SECTION B -- Analytical Services

Please respond to the following questions concerning the analysis of your samples.

1. Overall, how would you rate the CRL analytical services you received?

   Bad ___ ; Poor ___ ; Fair ___ ; Good ___ ; Excellent ___

2. If not "Good" or "Excellent", what was the problem?

3. What type of analytical services did you request (eg, analysis of samples, etc.; lab audit; document review, other)?

4. Who performed the analytical service(s) (CRL EPA Staff, ESAT)?

## SECTION C -- Comments and Suggestions

Please provide specific comments or suggestions for improving any of the aspects of CRL Analytical Services:

If you would like additional information on CRL Analytical Services, The CRL Board of Directors, or the Sample Request Process, please indicate below (✓) and provide your name and mail code).

Analytical Services ____; CRL Board of Directors _____; Sample Requests _____

Name: _____ Mail Code: _____

**FORM SUBMISSION**

***Thank you*** for taking the time to answer the questions in our survey. You will receive a confirmation message from us shortly.

We will review your survey and respond to any specific concerns or problems ASAP. Your survey and others will be evaluated for trends in an effort to establish efficient support and analytical processes. The process at each stage of the analytical services we provide are critical links towards giving you the kind of timely, accurate analytical services you need. This data will also be tracked by our management and the Board of Directors so additional customer feedback can be used to plan CRL activities in the future.

Please forward this completed survey to:

**CRL Sample Coordinator** at **Mail code: ML-10C**

Please go to the following e-mail address at: schupp.george@epa.gov to request an electronic copy of this survey or call 312-353-1226.

# CRL Data Review Qualification Codes

| QUALIFIER | DESCRIPTION |
|---|---|
| **B** | This flag is used when the analyte is found in the associated ***B***lank as well as the sample. It indicates possible blank contamination and warns the user to take appropriate action while assessing the data. See the case narrative for a discussion of common lab contaminants and/or the relative concentration of contamination in the samples and blanks for relevance. |
| **J** | This flag is used when the analyte is estimated due to quality control limit(s) being exceeded. This flag accompanies all GC/MS tentatively identified compounds (TICs). This flag also applies to a suspected, unidentified interference. This flag is placed on affected detected results as well as non-detected (i.e., "U" flagged) results. (***J*** is the flag used in the Superfund CLP SOW and Data Review Functional Guidelines and is used by CRL for consistency.) |
| **M** | This flag is used when the analyte is confirmed to be qualitatively present in the sample, extract or digestate, with a quantity at or above the CRL ***M***ethod Detection Limit (MDL) but below the lowest concentration of the calibration curve. This flag indicates the quantitated value is estimated since it falls below the lowest calibration standard in the calibration curve. |
| **N** | This flag applies to GC/MS Te***N***tatively Identified Compounds (TICs) that have a mass spectral library match. |
| **Q** | This flag applies to analyte data that are severely estimated due to quality control and/or ***Q***uantitation problems, but are confirmed to be qualitatively present in the sample. No value is reported with this qualification flag. |
| **R** | This flag applies to analyte data that are ***R***ejected and unusable due to severe quality control, quantitation and/or qualitative identification problems. No other qualification flags are reported for this analyte. No value is reported with this qualification flag. |
| **U** | This flag in used when the analyte was analyzed for but ***U***ndetected in the sample. The CRL RL for the analyte accompanies this flag. When the customer requests CRL to report below our RL down to our MDL, undetected analytes are reported with a "U" code and the MDL. As with sample results that are positive, the value is corrected for dry weight, dilution and/or sample weight or volume. |

**Date:** 10 August 2001
**Analyst:** John V. Morris
**Sample Batch Number:** 20010051&20010052
**Facility Name:** Cheshire Monitoring Study
**Analyte**: ICP Metals

**Narrative for the Analysis of Metals in Water in Batches 20010051&52**

On 3 August 2001, two batches of air filters, comprising four filters each (in 20010051: 2001AH01S01, station ID GHS; 2001AH01D01, station ID GHS; 2001AH01S02, station ID RVHS; 2001CM01S03, station ID ADDAVILLE; in 20010052: 2001AH02S01, station ID GUIDING HANDS; 2001AH02D01, station ID GUIDING HANDS; 2001AH02S02, station ID RVHS; and 2001AH02S03, station ID ADDAVILLE) were received at CRL for the analysis of metals. The batch numbered 20010051 was collected on 24 July 2001 and the batch numbered 20010052 was collected on 30 July 2001. The analysis was limited to the metals listed on page 15 of the QAPP (attached).

The samples were prepared on 6 August 2001. Method Metals_006, a hot block adaptation of the beaker digestion given in 40 CFR Part 50, Appendix G, was used for the digestion. The digestion log number was 1291. There are no holding times for the air program. This analyst neglected to cut duplicate filter strips from one filter from each batch. These will be cut and analyzed with the next batch of filters from this study.

Three filter blanks were taken from the same lot as the filters used in this study. As can be seen, the barium, chromium, iron and magnesium results on the filters were significant relative to the results measured for the exposed filters. Also, one nickel result was above reporting limit. Due to the contribution from the filter material itself for these metals, the data for all metals are presented as μg/filter. The client should examine the data in light of the needs of the study and determine the value of the data for these elements. Data in μg/filter may be divided by the air volume to arrive at data expressed as $\mu g/m^3$.

The analysis was performed on 7 August 2001 using method Metals_003, using the Perkin-Elmer 3300DV ICP. The yttrium internal standard readings were consistent throughout the run.

For the thirteen metals reported for this study, all instrument check standards (LCM1, LCM2, Hi AQC) were in control, except for the first cadmium LCM1 (111% recovery). This affected only the cadmium results for the report level check (RLC) and the spectral interference check (SIC) solutions. For blanks, beryllium, copper and magnesium were the only reported elements with flags on the instrument blank (LCB). For beryllium, the last LCB was just slightly more negative than the MDL, but the sample results were well below the reporting limit, so the data was not flagged. For copper and magnesium, the data was all much higher than the reporting limit, so the data was not flagged. For the digestion blank, copper, lead and magnesium were outside the limits of ± MDL, but either the data were much greater (copper and magnesium) or

**Date:** 10 August 2001
**Analyst:** John V. Morris
**Sample Batch Number:** 20010051&20010052
**Facility Name:** Cheshire Monitoring Study
**Analyte:** ICP Metals

the difference of the data and the blank was not enough to indicate a false negative for lead, with the exception of samples 2001AH02D01 and 2001AH02S02. These two samples are given a "J" flag because the results would have been reportable had the amount of the negative blank (corresponding to about -0.4 µg/filter) been added. For copper, the RLC was not recovered at all, but the difference between the RLC result (-0.001 mg/L) and the instrument blank (-0.005 mg/L) was just the RLC concentration. This problem with the copper blank was attributed to some residual copper giving a false signal on the calibration blank. As stated above, the copper data were all much higher than the blank, so the data were not flagged. Spike recoveries for both the spiked blank (LFB) and the spiked filter blank are within the expected 100±15%. All the SIC solutions show no problems for these samples, as the concentrations of any interfering species are quite low.

The printer jammed near the end of the run, so a duplicate copy of the raw data was printed from the results file. Both copies are included in the review package.

All analytical results files, sample information files and reformat files for ICP analysis can be found on the R5CRL data server using the following path:
h:\r5crl\vol3\metals\jvmorris\20010051_52\3300dv\

The narrative, QC summary spreadsheets, sample result calculation spreadsheets and the final sample report for ICP analysis can be found on the R5CRL data server using the following path:
h:\r5crl\vol3\metals\jvmorris\20010051_52\reports\

**List of Pollutants to be Analyzed by USEPA Central Regional Laboratory**

- Arsenic
- Barium
- Beryllium
- Cadmium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Magnesium
- Manganese
- Nickel
- Selenium

**Other Possible Pollutants to be Analyzed by USEPA Central Regional Laboratory**

- Sulfates
- Nitrates
- H2SO4

## 2.5 Quality Control

The quality control checks for the PM10 and TSP sampler flow rate calibration will be performed at least monthly. Deviations of greater than ± 7% of the audit flow as compared to the sampler calibration relationship will require recalibration of the sampling device. Deviations exceeding ±10% from the design flow rate will be investigated and may result in invalidation of all data obtained subsequent to either the last acceptable calibration or the last acceptable audit.

Internal quality control checks are necessary for the filter preparation and gravimetric filter analysis procedure. Specific details (See Appendix B) will follow guidelines listed below:

- Filter weighing specifications require that weighing room temperature is to be 15-30 degrees C and held to ±3 degrees C.

- Relative humidity of the weighing room is to be 20-45% RH and constant within ±5%.

- Balances used for weighing PM10 filters will be checked with Class S weights between 3g and 5g. Actual and measured weights must agree within ±0.5 mg. Zero QC checks must be within ±0.5 mg of true zero.

- Reweighing of exposed filters should agree with original weights within ±5.0 mg. 10% (or at least one filter per weighing session) of exposed filters will be reweighed.

US EPA CRL - Region V
ICP Final Report Results
Air Filters

| | | | |
|---|---|---|---|
| Sample Number: | 2001AH02S01 | Station ID: | GUIDING HANDS |
| Sample Batch Number: | 20010052 | Study: | Cheshire Monitoring Study |
| Analysis Date: | 7 Aug 01 | | |

| Element | Concentration | | Units |
|---|---|---|---|
| Arsenic | 9 | U | µg/filter |
| Barium | 145 | | µg/filter |
| Beryllium | 0.6 | U | µg/filter |
| Cadmium | 0.6 | U | µg/filter |
| Chromium | 5.66 | | µg/filter |
| Cobalt | 1.2 | U | µg/filter |
| Copper | 815 | | µg/filter |
| Iron | 363 | | µg/filter |
| Lead | 10.9 | | µg/filter |
| Magnesium | 656 | | µg/filter |
| Manganese | 81.3 | | µg/filter |
| Nickel | 2.67 | | µg/filter |
| Selenium | 20.5 | | µg/filter |

Jun
10 Aug 01

US EPA CRL - Region V
ICP Final Report Results
Air Filters

| | | | |
|---|---|---|---|
| Sample Number: | 2001AH02D01 | Station ID: | GUIDING HANDS |
| Sample Batch Number: | 20010052 | Study: | Cheshire Monitoring Study |
| Analysis Date: | 7 Aug 01 | | |

| Element | Concentration | | Units |
|---|---|---|---|
| Arsenic | 9 | U | µg/filter |
| Barium | 144 | | µg/filter |
| Beryllium | 0.6 | U | µg/filter |
| Cadmium | 0.6 | U | µg/filter |
| Chromium | 5.17 | | µg/filter |
| Cobalt | 1.2 | U | µg/filter |
| Copper | 676 | | µg/filter |
| Iron | 347 | | µg/filter |
| Lead | 6 | U,J | µg/filter |
| Magnesium | 631 | | µg/filter |
| Manganese | 75.5 | | µg/filter |
| Nickel | 2.50 | | µg/filter |
| Selenium | 22.5 | | µg/filter |

Jm
10 Aug 01

US EPA CRL - Region V
ICP Final Report Results
Air Filters

| | | | |
|---|---|---|---|
| Sample Number: | 2001AH02S02 | Station ID: | RVHS |
| Sample Batch Number: | 20010052 | Study: | Cheshire Monitoring Study |
| Analysis Date: | 7 Aug 01 | | |

| Element | Concentration | | Units |
|---|---|---|---|
| Arsenic | 9 | U | µg/filter |
| Barium | 142 | | µg/filter |
| Beryllium | 0.6 | U | µg/filter |
| Cadmium | 0.6 | U | µg/filter |
| Chromium | 7.10 | | µg/filter |
| Cobalt | 1.2 | U | µg/filter |
| Copper | 150 | | µg/filter |
| Iron | 376 | | µg/filter |
| Lead | 6 | U,J | µg/filter |
| Magnesium | 660 | | µg/filter |
| Manganese | 61.3 | | µg/filter |
| Nickel | 3.62 | | µg/filter |
| Selenium | 47.7 | | µg/filter |

JWM
10 Aug 01

US EPA CRL - Region V
ICP Final Report Results
Air Filters

| | | | |
|---|---|---|---|
| Sample Number: | 2001AH02S03 | Station ID: | ADDAVILLE |
| Sample Batch Number: | 20010052 | Study: | Cheshire Monitoring Study |
| Analysis Date: | 7 Aug 01 | | |

| Element | Concentration | | Units |
|---|---|---|---|
| Arsenic | 9 | U | µg/filter |
| Barium | 174 | | µg/filter |
| Beryllium | 0.6 | U | µg/filter |
| Cadmium | 0.6 | U | µg/filter |
| Chromium | 8.38 | | µg/filter |
| Cobalt | 1.2 | U | µg/filter |
| Copper | 86.9 | | µg/filter |
| Iron | 352 | | µg/filter |
| Lead | 6 | U | µg/filter |
| Magnesium | 743 | | µg/filter |
| Manganese | 56.2 | | µg/filter |
| Nickel | 3.16 | | µg/filter |
| Selenium | 95.8 | | µg/filter |

10 Aug 01

Date: August 9, 2001
Reviewer: Marilyn Jupp
Sample Batch Number: 20010051 & 20010052
Facility Name: Cheshire Monitoring Study
Analyte: ICP Metals on Air filters

Please move the paragraph discussing the blank filter contamination to immediately after the date and type of analysis paragraph. These findings have the greatest impact on the usefulness of the results and should be mentioned early.

Please state that these results are being reported in ug/filter, rather than the usual ug/m3, because of high blank filter values. The air filter preparation SOP states that an average and standard deviation will be provided for the blank filter results when they are reported in ug/filter. Please add these calculated values to the results.

Please define RLC and SIC in your narrative. RLC is not, that I could find, defined under QC in any of the ICP methods. Codes should be defined in the narrative in any case. The definition was found only in the result report.

Concerning beryllium, please say "but the sample results were **well below** the reporting limit, so the data was not flagged." I took the original statement of "not close" to mean that the results were well **over** the reporting limit.

Concerning lead, the negative differential in the blank calculates to 0.4 ug/filter. Sample 2001AM02D01 yields a value of 5.8 and 2001AM02S02, 5.95. These samples would have been above the reporting limit with the addition of the 0.4 ug. Please state that lead has a negative bias and that these two samples are near enough to the reporting limit that the true value might be above the reporting limit.

The sample identifications in the reports are, for example, 2001AM02D01; the sample identifiers in the raw data are 2001AH02D01. Perhaps this should be mentioned as an error.

CRL Metals Data Review Checklist

Batch Number: 20010051852 Facility: Cheshire Monitoring Study

Parameter: ICP (short list)

| Package Overview: | Review: | |
|---|---|---|
| | Analyst | Peer |
| Raw Data Package Complete? | ✓ | ✓ |
| Results Reported Correctly? | ✓ | ✓ |
| Special Requests Done? | ✓ | ✓ |
| Calculations Checked? | ✓ | ✓ |
| Calibration Not Exceeded? | ✓ | ✓ |
| Field QC Checked? | ✓ | ✓ |
| **Quality Control:** | | |
| Holding Times Met? | NA | NA |
| Preservation Checked? | NA | NA |
| Proper Digestion Verified? | ✓ | ✓ |
| Initial Instrument Performance Checks Verified? | ✓ | ✓ |
| Calibration Verification Checked? | ✓ | ✓ |
| Sample-Specific QC (Internal Standards or Analytical Spikes) Okay? | ✓ | ✓ |
| Matrix QC Checked? no duplicates run! | ✓ | ✓ |
| Digestion Blanks Checked? | ✓ | ✓ |
| Spiked Blank Checked? | ✓ | ✓ |
| LCS (if applicable) Checked? | NA | NA |
| Species QC (if applicable) Checked? | NA | NA |
| **Final Check:** | | |
| Technical Review Done? | ✓ | ✓ |
| Narrative Complete? (See sect. 7 of data review SOP, HK005) | ✓ | ✓ |

Analyst: [signature] Date: 8 Aug 01

Peer Reviewer: [signature] Date: 8/9/01

CRL Form Version 03.1/00

Comments Attached? (Y/N) Y